(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) Publication number: **0 394 689 A2**

(12) EUROPEAN PATENT APPLICATION

(21) Application number: **90105915.4**

(51) Int. Cl.5: **D06M 15/643**

(22) Date of filing: **28.03.90**

(30) Priority: **29.03.89 US 330108**

(43) Date of publication of application:
31.10.90 Bulletin 90/44

(84) Designated Contracting States:
AT BE CH DE ES FR GB GR IT LI LU NL SE

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(54) Method of treating fabrics and other substrates with exhaustible cationic silicones.

(57) A method of improving the softness, or antistatic properties of a textile without yellowing comprising treating said textile with a composition comprising a) about 0.05 to about 5 weight percent of a cationic silicone of the structure $M\ D_x\ D'_y\ M$ where M is $(CH_3)_3\ SiO_{1/2}$; D is $OSi(CH_3)_2$; D' is $CH_3SiO(CH_2)_3OCH_2CH(OH)CH_2N^+(CH_3)_3Cl^-$; x = 20-200; y = 3-60; and b) about 0 to 2 % of an electrolyte.

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METHOD OF TREATING FABRICS AND OTHER SUBSTRATES WITH EXHAUSTIBLE CATIONIC SILICONES

BACKGROUND OF THE INVENTION

This invention relates to methods of treating fabrics and other substrates with exhaustible cationic silicone compounds which impart improved softness and antistatic properties to the treated fabric without yellowing.

Fabrics have been treated for many years to improve their properties and acceptability to consumers. Improved softness, reduction in static, greater ease in ironing and prevention of yellowing are among the desired effects. Some treatments used to achieve these results, however are counter productive. For example, application of durable press resins to improve ease of ironing is known to give fabrics a harsher hand.

It has been well known to apply various types of textile conditioners to make textiles softer, and reduce static. Among the conditioners used have been nonionic silicones, cationic emulsions of nonionic silicones and organic quaternary compounds.

Nonionic silicones such as dimethyl silicone oil, silanol silicone fluids, silanic hydrogen fluids and an epoxy functional silicone available from Union Carbide Corporation under the tradename UCARSIL® T-29, have been used as textile softeners for a number of years. These materials, however, have no particular affinity for the textile substrate. Therefore, they are applied only by physical contact with the textile. These silicones are applied mostly by pad bath techniques because they are not exhaustible, i.e. because of their lack of affinity for the textile substrate they are not adsorbed from the pad bath solution onto the textile substrate.

Exhaustible organic quaternary softeners are known such as methyl-1 (tallow amido ethyl) 2-(tallow) imidazolinium methyl sulfate, dimethyl alkyl(C_{12} - C_{16}) benzyl ammonium chloride available from Sherex Chemical Co. Corp. under the tradenames Varisoft 475® and Variquat 50 MC®, respectively, and N-cetyl-N-ethyl morpholinium ethosulfate available from Atlas Chemical Co. under the tradename Atlas G-263®, but they are deficient in their softening properties and tend to yellow the textile substrate, especially when white or pastel fabrics are used.

Use of quaternary ammonium functional silicones to treat fabrics is also known in the art. For example U.S. - A -4,384,100, 4,511,727 and 4,615,706 concern quaternary ammonium functional silicone compounds prepared by reacting carboxylic acid functional quaternary ammonium compounds with carbinol functional silicon compounds. These materials are claimed to improve the antistatic properties of the fabric, however no mention is made of improved fabric softness or non-yellowing properties.

Similarly U.S. - A - 4,390,713 and 4,394,517 disclose processes for preparing quaternary ammonium-functional silicon compounds by reacting carbinol functional quaternary ammonium compounds with carboxylic acid-functional silicon compounds and by reacting carboxylic acid functional quaternary ammonium compounds with amino functional silicon compounds, respectively. Both patents assert that the resultant cationic silicones are useful as antistatic finishes for textiles. No mention is made in either patent of softness, non-yellowing or exhaustability improvements.

U.S. - A - 4,417,066 discloses a process for preparing organosiloxane polymers, which are useful as soil release agents, made by reacting a silanol terminated polydiorganosiloxane and a quaternary ammonium silane. No yellowing or exhaustability improvements were noted.

U.S. - A - 4,448,810 discloses the use of a polydiorganosiloxane containing at least one quaternary ammonium salt substituent which is used as a soil release agent and to impart an antistatic finish. An additional siloxane is added to provide soft hand, lubricity or recovery from creasing. No mention is otherwise made of improved softness, yellowing properties or the ability to exhaust from solution.

U.S. - A - 4,614,675 discloses a composition for treating a solid material to give it antimicrobial, hydrophilic and antistatic properties comprising a siloxane compound which has one or more alkoxy silylalkyl groups and one or more polyoxyalkylene groups and a silane having antimicrobial properties. No improvements in hand, yellowing properties or the ability to exhaust from solution is disclosed.

U.S. - A - 4,585,563 discloses a detergent composition containing an organosiloxane that can have quaternary functionality, to improve softness.

German Offenlegungsshrift DE 3542725 discloses a composition comprising an aqueous mixture of a cationic silicone oil, a cationic fatty acid condensate, and a cationic film former for treating laundry added to the final rinse to facilitate ironing.

U.S. - A - 4,504,541 discloses the use of quaternary monomeric silicon structures as fabric antimicrobial

treatments where the quaternary ammonium cation is at least partially sealed with an anionic surfactant. Improved susceptibility to discoloration is also disclosed, however no mention is made of fabric softening.

U.S. - A - 4,767,547 discloses a rapidly biodegradable fabric softening composition which may contain a silicone component. Cationic silicones are preferred.

GB - 1,549,180 discloses a fabric treatment composition containing a cationic compound, and an emulsion containing a cationic siloxane compound.

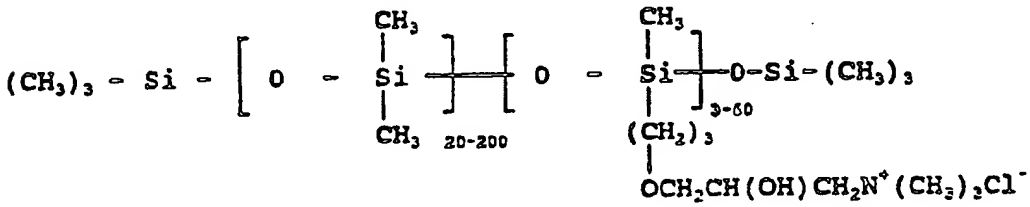
None of the above references disclose the cationic silicones used in the present invention.

GB - 2,201,696 and 2,201,433 disclose quaternary silicones for use in fabric conditioning compositions to provide improved wettability and softness. While the silicones broadly disclosed in these applications appear to include the silicones used in the present invention, the compounds specifically disclosed therein are shown to be no better than organic softeners in improving textile softness.

The cationic quaternary ammonium compounds with the structure used in the present invention were previously known and are described in U.S. - A - 3,389,160 where they were described as being useful as surfactants, surface tension depressants and corrosion inhibitors. Cationic silicone compounds of the same class as used in the present invention were also described as being useful hair conditioners in U.S. - A - 4,185,087.

SUMMARY OF THE INVENTION

We have discovered that cationic silicones represented by the general structure:



which may also be represented by the following structure:

$M D_x D'_y M$

where M is $(CH_3)_3 SiO_{1/2}$;

D is $OSi(CH_3)_2$;

D′ is $CH_3SiO(CH_2)_3OCH_2CH(OH)CH_2N^+(CH_3)_3Cl^-$;

x = 20-200; and

y = 3-60

efficiently exhaust (i.e., are adsorbed from solution) onto a variety of fabrics. These compounds impart an unexpected high level of soft, silky hand to the fabrics without yellowing them. In addition, these materials have been shown to exhibit antistatic properties under low relative humidity conditions (30% R.H).

We have further found that in the more preferred embodiments our invention comprises treating fabrics with a bath containing 0.05-5.0% by weight, based on the total fabric weight, of cationic silicone solids of the formula $MD_{20-200}D'_{3-60}M$ together with 0-2% by weight, based on the total bath weight, of an electrolyte such as NaCl, KCl, Na_2SO_4 or $MgSO_4$. Ideally, 0.1 to 10 percent by weight, based on the weight of the fabric, of the cationic silicone is adsorbed by the textile.

Alternatively, the cationic silicones may be applied to a solid substrate in an amount of 0.1-10% by weight of said substrate so that the cationic silicones can be imparted to textiles by placing the substrate with clothes or fabrics in a dryer.

As optional ingredients, the fabric treatment composition may also include other fabric treatment agents known to those skilled in the art.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

CATIONIC SILICONES

The essential component of the present invention is a cationic silicone of the general structure
$MD_x D'_y M$
where M is $(CH_3)_3 SiO_{1/2}$;
D is $OSi(CH_3)_2$;
D' is $CH_3SiO(CH_2)_3OCH_2CH(OH)CH_2N^+(CH_3)_3Cl^-$;
x = 20-200; and
y = 3-60.

Suitable cationic silicones according to the present invention may be either water soluble, water dispersible or water insoluble.

Both x and y segments of the cationic silicone are repeated randomly throughout the molecule. It is preferred that x is more than 20, because when x is less than 20 the improvements in fabric hand of this invention are marginal. It is also preferred for practical reasons that x not exceed 200, because when x exceeds 200, the resultant cationic silicone is much more difficult to handle and emulsify. Y is preferably greater than three to ensure the compound has sufficient cationic character to achieve the exhaustability benefits of this invention.

Examples of water soluble cationic silicones to be used in accordance with the present invention are $MD_{30}D'_{30}M$ and $MD_{165}D'_{50}M$. The water soluble cationic silicones have enhanced antistatic properties. However, the non-water soluble cationic silicones are preferable to the water soluble compounds because they provide even greater improvements in softness. A preferred water insoluble cationic silicone compound is $MD_{165}D'_{16.5}M$. The most preferred is $MD_{150}D'_{10}M$.

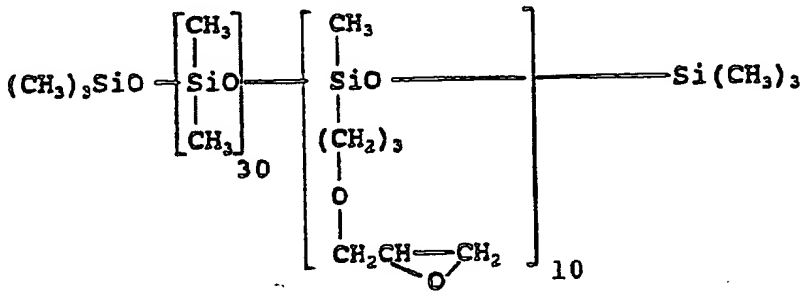
The cationic silicones of the present invention may be synthesized by methods known in the art, as disclosed, for example in U.S. - A - 3,389,160, whose disclosure is incorporated by reference herein.

As an example of how the cationic silicones of the present invention may be prepared follows:

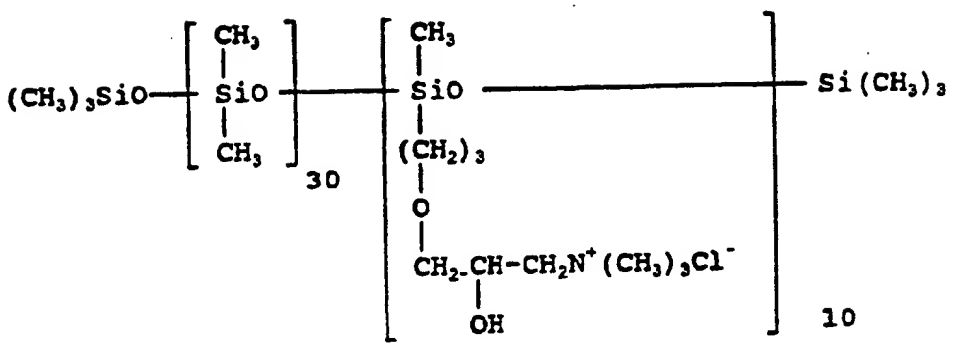
A 500 ml three-neck flask was equipped with a stirrer, addition funnel, dry ice/acetone condenser, thermocouple and electric heating mantle. A nitrogen blow-by was placed on the outlet from the dry ice/acetone condenser.

A solution of trimethylamine in isopropanol was prepared by sparging the amine through the isopropanol. The resulting solution had an amine concentration of 0.0027 milliequivalents per gram.

The flask was charged with 18.6 g of trimethylammonium chloride (0.195 equivalent) and 9.05 g of isopropanol was added and stirred. Then 68.9 g of the trimethylamine / isopropanol solution (0.000186 trimethylamine equivalent) was added with moderate agitation. To the stirred solution there was rapidly added 77.95 g of the epoxy pendant siloxane copolymer of the formula $MD_{30}D''_{10}M'$ (0.192 oxirane equivalent) over a five minutes period having an epoxy ring content of about 2.53 milliequivalents per gram and the structure:



The contents were stirred and heated to 80° C; about 5 minutes after this temperature was reached all of the trimethylammonium chloride had gone into solution. After heating and stirring at 80° C for 4.5 hours the flask was cooled and sparged overnight with dry nitrogen. The quarternary ammonium pendant siloxane copolymer produced had the formula $MD_{30}D'_{10}M$



A 61.66 g portion of the reaction product mixture and 20 g of propylene glycol were charged to a rotovap and stripped at 45° C at a nine speed for 1.5 hours to remove isopropanol and other low-boiling materials. In this example the ratio of free tertiary amine equivalent to tertiary amine acid salt equivalent is 0.001:1.

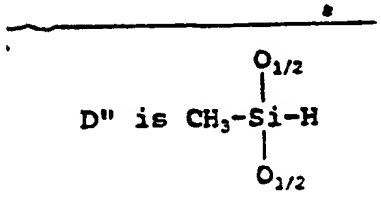
The most preferred compound $MD_{150}D'_{10}M$ of Applicants' present invention may be made by the following procedure:

Preparation of Silanic Hydrogen Fluid

1. Components

	Wt.%	
$MD''_{40}M^{1/}$	5.32	
Octamethylcyclotetrasiloxane	93.54	
$(CH_3)_3$-Si-O-Si-$(CH_3)_3$	1.14	
H_2SO_4 (conc.)	0.82	ml
$NaHCO_3$	4.5	g

2. Procedure



D'' is CH_3-Si-H with $O_{1/2}$ above and below

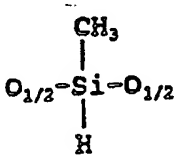
$MD''_{40}M$, octamethylcyclotetrasiloxane and $(CH_3)_3$-Si-O-Si-$(CH_3)_3$ were charged to a 3-neck flask equipped with a mechanical stirrer, condenser, and nitrogen purge. Concentrated sulfuric acid was added and stirred for 24 hours. $NaHCO_3$ was added and neutralized for 2 hours. The product was then filtered.

3. Properties of SiH Fluid

Viscosity (Oswald, mm^2/s: 253.6 - 279.6
SiH Content (cm^3 H_2/g): 18.9 - 19.5 ± 0.6 (theory 18.9)

Preparation of Allylglycidylether Intermediate

Eighty pounds of isopropyl alcohol was charged to a 100 gallon reactor with agitation. Twenty-six pounds of allyl glycidyl ether was then added to the reactor followed by 215 pounds of the $MD_{150}D''_{10}M$ produced in the preceding reaction (where D'' is

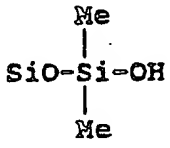


and M and D are as previously defined), and 0.18 kg (0.4 lbs) of sodium propionate as a buffer. At this point the solvent was checked to verify that the pH is 6.5-7.5 and the % water was less than 0.1%. The solution is then heated to 60° C. Once 60° C was reached, the kettle temperature control was set at 50° C. When the temperature of the solution began to fall, 80 cm^3 of 10% chloroplatinic acid was added, resulting in a 5 to 7 degree exotherm.

The kettle contents were maintained at 75° C for 2 hours. After 3 hours it was verified that SiH was below 0.2 cm^3 H_2/g.

Quaternization

An additional 12.6 kg (28 lbs) of isopropyl alcohol were charged to the reaction kettle. 21 g (0.046 lbs) of triethylamine then was charged into the kettle followed by 9.9 kg(22 lbs) of trimethylamine hydrochloride. The contents were heated to 75° C and held for 3 hours. After 3 hours, the percentage epoxy and solvent pH are checked to see if the % epoxy was no more than 0.1%. Water was then added to the reactants, and the kettle contents were neutralized with 0.5 weight percent glacial acetic acid. The product is then filtered, resulting in a product with 67% solids in isopropanol with the formula $MD_{141}D^*_{12.5}D^1_{0.5}M$ wherein D^1 has the formula



and a viscosity of 244 mm^2/s.

The resulting cationic silicone/isopropanol formulation contains a target of 69-71% of silicone solids. The cationic silicone has a targeted viscosity of 800-1,000 mm^2/s and a pH of 5-7.

EMULSIONS

The water soluble cationic silicones of the present invention may be directly incorporated into the medium used to treat the textiles, in an amount sufficient to deposit 0.1 to 10 percent by weight of the cationic silicone on the textile, based on the weight of the textile.

When the cationic silicones to be used in the present invention are water insoluble, it is preferred that they be put into emulsion form. Any number of emulsifiers may be used including, but not limited to alkanolamides, aklylaryl sulfonates, amine oxides, sulfonated and/or ethoxylated amines and amides, betaine derivatives, carboxylated alcohol ethoxylates, ethoxylated alcohols (primary or secondary) ethoxylated alkyl phenols, ethoxylated fatty acids, ethoxylated fatty esters and oils, fatty acid esters, glycerol and glycol esters, imidazolines and imidazoline derivatives, isethionates, olefin sulfonates, phosphate esters, and alkylaryl quaternary ethosulfates.

Applicants do not suggest that any emulsifier will necessarily produce an acceptable emulsion with any cationic silicone. Applicants note that some difficulty in preparing stable emulsions was encountered using the following emulsifiers with $MD_{150}D'_{10}M$ and the emulsification procedure specifically described herein:

Tradename	Manufacturer	Chemical Name
Varisoft 222®	Sherex Chemical Co. Corp.	complex di-fatty
Varisoft 238®	Sherex Chemical Co. Corp.	quarternary surfactants
Variquat 200®	Sherex Chemical Co. Corp.	trimethyl benzyl ammonium chloride
Arquad 2HT75®	AKZO Chemie America, Armac Chemical	di-hydrogenated tallow dimethyl ammonium chloride
Adogen 442®	Sherex Chemical Co. Corp.	dimethyl hydrogenated tallow ammonium chloride
Atlas G-271®	Atlas	N-soya-N-ethyl-morpholinium ethyl sulfate

However, it is believed that one skilled in the art, using conventional emulsification techniques, such as application of heat, regulation of order of addition of ingredients, and proper selection of mixer speeds and homogenizer pressures can obtain emulsions with the emulsifiers disclosed herein.

The emulsifiers used may be nonionic or cationic. Preferred nonionic emulsifiers are TERGITOL® 15-S-15 and TERGITOL® 15-S-3 which are secondary alcohol ethoxylates available from Union Carbide, and Tween 80, polyoxyethylene sorbitan monooleate, available from ICI, Americas. Preferred cationic emulsifiers are Atlas G-263®, morpholinium ethosulfate, and Varisoft 475® (Sherex) which is methyl 1 -(tallow amido ethyl) 2-(tallow) imidazolinium-methyl sulfate.

Typically, an emulsion for use in accordance with the present invention will contain 2 to 80 parts by weight of a cationic silicone formulation, said formulation containing 70 parts by weight silicone solids in an inert solvent such as isopropanol. Other suitable inert solvents include ethanol, methanol and butanol. Preferably the emulsion contains 5 to 40 weight parts by weight of the cationic silicone formulation, and most preferably 10 to 25 parts by weight of the cationic silicone formulation.

The emulsion is made using an emulsifier in a quantity amounting to 5 to 40% by weight of the amount of cationic silicone formulation used. Preferably, the emulsifier is used in an amount of 10-30% by weight, and most preferably about 15-25% by weight of the amount of cationic silicone formulation used. The balance of the composition is of course, water.

A preferred emulsion made with non-ionic emulsifiers contains 14.3 parts of $MD_{150}D'_{10}M$ formulation (70% active silicone in isopropanol and water), 1.8 parts TERGITOL 15-S-15®, 1.2 parts TERGITOL 15-S-3® and 82.7 parts water. All parts are by weight.

The emulsion may contain other additives as desired, including antimicrobial agents, antifoam agents, as well as other silicones and organic softeners.

A preferred cationic emulsion is made with 14.3 parts $MD_{150}D'_{10}M$ (70% actives, balance isopropanol and water), 81.5 parts water and 4.2 parts Atlas G-263®. Again, all parts are by weight.

Any suitable means known in the art for preparing the emulsions may be used. Using the preferred cationic emulsion composition described above as an example, one preferred emulsification method involves adding the emulsifiers to the cationic silicone followed by mixing at moderate speed for 10 minutes. 15 parts of the water is then charged to the mixture followed by another 10 minutes of mixing at moderate speed. Ten additional parts of H_2O are added with another 10 minutes of mixing thereafter. The balance of the water is then added followed by additional mixing for 10 minutes. The speed of the mixer is reduced as the emulsification nears completion.

In those instances where the chosen emulsifier is solid at room temperature, it is recommended that it be heated to a fluid state prior to its addition to the cationic silicone component.

The cationic silicones may be applied in accordance with the present invention to any suitable textile, including, but not limited to wool, cotton, rayon, hemp, natural silk, polypropylene, polyethylene, polyester, polyurethane, polyamide, cellulose acetate, polyacrylonitrile fibers, and mixtures of such fibers. The textile materials may consist of staple fibers or monofilaments. The fabrics may be knits, weaves, papers and other non-wovens, resin finished fabrics and their sewn products.

The cationic silicones may be applied in the textile manufacturing process by any suitable method known in the art including but not limited to pad baths, spraying, foam finishing, minimum application, screen printing, sizing baths, etc. The preferred methods of application are by pad bath and exhaustion

from solution processes.

Other textile treatments may be applied in conjunction with the cationic silicone including, but not limited to optical brighteners, soil release agents, water repellents, durable press resins, other softeners, or even dyes.

It is known, that antimicrobial properties may be imparted by application of cationic quaternary compounds to various substrates. Application of the cationic silicones of the present invention can also impart antimicrobial activity to the textile substrate.

The present invention is also suitable for use by consumers. For example, cationic silicones compositions may be added to the wash or rinse cycles in the laundry. Or they may be absorbed onto a solid substrate and applied to clothes in the dryer. The cationic silicone could also be pre-blended with detergent and added to the washing machine.

As shown in the following examples of the present invention, the presence of inorganic electrolyte can in some cases enhance the exhaustion of the cationic silicones. Ideally, the fabrics to be treated are contacted with a composition containing about 0.05 -2% of the cationic silicone component and about 0-2% of an electroyte such as NaCl, KCl, Na_2SO_4 and $MgSO_4$. About 0.1 to about 10% by weight of the cationic silicone, based on the weight of the textile, will be adsorbed by the textile.

The following examples demonstrate the improved softness, anti-yellowing, antistatic and exhaustability characteristics provided by practice of the present invention. They are provided to more clearly illustrate the invention, and are not to be construed as limiting.

The improved softening of the present invention is demonstrated in Example 1.

EXAMPLE 1

A series of cationic silicone polymers were prepared and emulsified if necessary.

Emulsions containing $MD_{150}D'_{10}M$ and $MD_{165}D'_{16.5}M$ were prepared using 20 parts of a cationic silicone formulation which contained 50% actives in isopropanol, 1.2 parts TERGITOL 15-S-15®, 0.8 parts TERGITOL 15-S-3®, and 78 grams water.

The $MD_{90}D'_{30}M$ and $MD_{165}D'_{50}M$ compounds are water soluble and were therefore added to the bath without emulsification.

A nonionic silicone emulsion, Ucarsil TE-24® available from Union carbide, which contains an epoxy functional silicone, was used as a standard.

The emulsions were applied to 100% cotton and 65/35 polyester/cotton by pad bath with and without 15 parts Intex FC510®, dimethylol dihydroxy ethylene urea (DMDHEU), a durable press resin and Intex FC58® a catalyst which enables cross-linking of the resin, such that 0.6% silicone solids were deposited on the fabric. The amount of silicone deposited on the fabric is determined by the analytical procedures described in G.W. Heylmun, et al., "Analysis of Methyl Fluorosilanes from Methypolysiloxanes by Gas Chromatography", J. of Gas Chrom. 1965, 266-268.

These materials were pad bathed onto the fabric because the silicone standard was nonionic and could not be exhausted onto the fabric. The fabrics were dry/cured at 171 ° C for 1.5 minutes to cure the durable press resin.

Softness Rating				
	65/35 = Polyester/Cotton Broadcloth		100% Cotton Print Cotton	
	No Resin	With Resin	No Resin	With Resin
Epoxy Functional Silicone Softener Standard	5	5	5	5
$MD_{150}D'_{10}M$	5	4	5.5	5.5
$MD_{165}D'_{16.5}M$	6	5.5	7	6.5
$MD_{90}D'_{30}M$	8.5	7	8	7
$MD_{165}D'_{50}M$	7	8	7.5	8
Untreated Fabric (no silicone)	9	8.5	9	9
1 = Softest; 5 = Standard; 10 = very harsh				

The fabrics were then subjectively evaluated for softness in blind tests with 5 panelists. A score of 5 is considered acceptable. A score of 6-7 is marginal and those of 8 and above are unacceptable.

EXAMPLE 2

The resistance to yellowing shown by fabrics treated in accordance with the present invention is shown in Example 2.

An emulsion containing $MD_{150}D'_{10}M$ was prepared using 20 parts of a cationic silicone formulation which contains 50% actives in isopropanol, 2 parts Varisoft 475® and 78 parts water.

The emulsified $MD_{150}D'_{10}M$ and an organic quaternary softener, Varisoft 475 which was simply dispersed in water, were separately applied to 65/35 = polyester/cotton and 100% cotton fabrics with or without the durable press resin DMDHEU, then dry/cured for one and a half minutes at 171° C. The silicone solids add-on level to the fabrics was 1.0 wt%.

When a durable press resin was used, the pad bath contained 15 parts of the durable press resin INTEX FC510® and 2.25 parts of INTEX Catalyst FC58.

To promote yellowing, the treated fabric samples were further exposed to heating for an additional 50 seconds or 100 seconds at 200° C. Whiteness values of the treatments were then measured according to AATCC Test Method 110-1979 (Reflectance, Blue, and Whiteness of Bleached Fabric).

WHITENESS VALUES OF FABRIC TREATED WITH CATIONIC SILICONE ($MD_{150}D'_{10}M$)

1% Silicone Solids (Based on Weight of Fabric) Pad Bath Application

I) 65/35 = Polyester/Cotton

	No DP Resin Fluorescence			With DP Resin Fluorescence		
		200° C			200° C	
	Initial	50 s	100 s	Initial	50 s	100 s
As Received	133	128	127	131	124	117
$MD_{150}D'_{10}M$ Silicone	132	128	124	130	122	117
Organic Quaternary (Varisoft 475)	129	127	122	127	119	113

II) 100% Cotton

	With DP Resin No Florescence		
		200° C	
	Initial	50 s	100 s
As Received	81	80	75
$MD_{150}D'_{10}M$	82	76	77
Organic Quaternary (Varisoft 475)	71	71	66

EXAMPLE 3

An emulsion containing ten percent actives of the cationic silicone $MD_{150}D'_{10}M$ was prepared using two parts Varisoft 475 per 10 parts silicone actives and 78 parts H_2O.

300 grams of water (adjusted to pH = 5.5 with acetic acid) was charged to a beaker and 0.73 g of the above emulsion was added. Two swatches of 100% cotton fabric (wt = 7.3 g were added to the beaker and

the beaker contents heated to 60°C (140°F) and held for 30 minutes while stirring. The level of silicone emulsion added to the beaker was such that 1.0% silicone solids would result on the fabric if 100% exhaustion was obtained.

A series of exhaustion studies such as described above was carried out wherein the electrolyte concentration (NaCl) in the water phase was varied from 0 to 1.5wt %. Although NaCl electrolyte was used in this example, other electrolytes such as KCl, Na_2SO_4, etc. could also be used. NaCl is preferred due to its low cost and ready availability.

The level of silicone deposited onto the fabric was measured by analyzing the treating solution by use of atomic absorption for silicon metal before and after the exhaustion process as well as by analyzing the level of silicone on the fabric before and after the exhaustion process.

In all cases (especially in the presence of salt in the water phase), the silicone treated fabrics after drying for 2 minutes at 110°C were much softer than the untreated control and the organic quaternary treated fabric.

The table below clearly shows that the presence of the electrolyte accelerates/enhances the exhaustion process for both the cationic silicone and organic quaternary. Further, less electrolyte may be used when dyed fabrics are treated.

EFFECT OF ELECTROLYTE CONCENTRATION ON EXHAUSTION EFFICIENCY	
(30 min. at - 60°C - (140°C)	
A) 100% Cotton Woven, White	
	% Exhaustion[2/]
$MD_{150}D'_{10}M$ (no NaCl)	8.8
$MD_{150}D'_{10}M$ + 0.25% NaCl	51.7
$MD_{150}D'_{10}M$ + 0.5% NaCl	68.3
$MD_{150}D'_{10}M$ + 1.0% NaCl	90.0
$MD_{150}D'_{10}M$ + 1.5% NaCl	96.8
Organic Quaternary (Varisoft 475)	7.7
Organic Quaternary + 1.5% NaCl	23.9
B) 100% Cotton Knit (Dyed Red)	
$MD_{150}D'_{10}M$	52
$MD_{150}D'_{10}M$ + 1% NaCl	91.5

2/ Based on analysis of solution before and after exhaustion process.

EXAMPLE 4

As described in Example 3, a variety of fabrics were utilized to demonstrate the broad exhaustible utility of the cationic silicone, $MD_{150}D'_{10}M$ in an emulsion made with Varisoft 475. In every case the softness properties of the treated fabric were markedly improved.

The fabric analysis records the amount of silicone on the fabric after treatment.

% CATIONIC SILICONE EXHAUSTED ONTO A VARIETY OF FABRICS, 1% NaCl IN WATER PHASE			
		Solution Appearance	
	Fabric Analysis	Before	After[3/]
Seersucker (50/50 = PE/C)	79%	hazy	clear
65/35 = PE/C, OES (prewashed) Woven	56%	cloudy	clear
50/50 = PE/C Knit (rose)	71%	hazy	clear
100% Cotton Terrycloth	93%	hazy	clear
100% Cotton Woven	90%	hazy	clear

3/ 30 min. at 60 °C (140 °F)

EXAMPLE 5

ANTISTAT PERFORMANCE

An emulsion containing $MD_{150}D'_{10}M$ was prepared using 20 parts of a cationic silicone formulation containing 50% actives in isopropanol, 2 parts Varisoft 475® and 78 parts water.

The emulsified cationic silicone $MD_{150}D'_{10}M$ and an organic quaternary VARISOFT 475® were pad bath applied to a variety of fabrics such that 0.6 wt% silicone or organic solids resulted on the fabric. The fabrics were dried for 1.5 minutes at 171 °C and conditioned at 30% relative humidity for 3 days. Antistat testing according to the test methods AATCC-76-1982 (Electrical Resistivity of Fabric) and FTMS4046-101C (Electrostatic Properties of Materials) were carried out.

	Surface Resistivity (ohms/square)	Decay Time (seconds)
65/35 = Polyester Cotton, Woven		
Cationic Silicone - $MD_{150}D'_{10}M$	5.5×10^{11}	1.7
*Organic Quaternary	1.4×10^{12}	6
Water	7×10^{12}	28
100% Cotton, Woven		
Cationic Silicone - $MD_{150}D'_{10}M$	4.4×10^{12}	25
*Organic Quaternary	4.0×10^{12}	20
Water	$>2 \times 10^{13}$	120
50/50 = Polyester/Cotton, Knit		
Cationic Silicone - $MD_{150}D'_{10}M$	1.8×10^{12}	4.4
*Organic Quaternary	3×10^{12}	6
Water	1.3×10^{13}	35

*VARISOFT 475® - Methyl-1 (tallow amido ethyl) 2-tallow imidazolinium methyl sulfate

These results show that the fabrics treated with the cationic silicone compounds had antistatic performance

better than or equivalent to that shown by the prior art organic quaternary compounds.

Claims

1. A method of improving the softness,or antistatic properties of a textile without yellowing comprising treating the textile with a composition comprising

a) about 0.05 to about 5 weight percent of a cationic silicone of the structure

$M\ D_x\ D'_y\ M$

where M is $(CH_3)_3\ SiO_{1/2}$;

D is $OSi(CH_3)_2$;

D' is $CH_3SiO(CH_2)_3OCH_2CH(OH)CH_2N^+(CH_3)_3Cl^-$;

x = 20-200;

y = 3-60; and

b) about 0 to 2 % of an electrolyte.

2. The method according to Claim 1 wherein the cationic silicone is water soluble or dispersible or not water soluble.

3. The method according to Claim 1 or 2 wherein the cationic silicone is $MD_{90}D'_{30}M$, $MD_{165}D'_{50}M$, $MD_{150}\ D'_{10}M$ or $MD_{165}D'_{16.5}M$.

4. The method according to at least one of Claims 1-3 wherein the cationic silicone is in an emulsion.

5. The method according to at least one of Claims 1-4 wherein the electrolyte in selected from NaCl, Na_2SO_4, $MgSO_4$ and KCl

6. The method according to at least one of Claims 1-5 wherein the textile contains a material selected from wool, cotton, rayon, hemp, natural silk, polypropylene, polyethylene, polyester, polyurethane, polyamide, cellulose acetate, polyacrylonitrile fibers and blends thereof.

7. The method according to at least one of Claims 1-6 wherein the treatment occurs during a textile manufacturing operation.

8. The method according to Claim 7 wherein the composition is applied by a technique selected from pad bathing, spraying, foam finishing, minimum application, screen printing and sizing baths.

9. The method according to at least one of Claims 1-8 wherein the treatment occurs during laundering or drying of the textiles.

10. The method according to at least one of Claims 1-9 wherein the composition includes additives selected from optical brighteners, soil release agents, water repellents, durable press resins, softeners and dyes.

11. The method according to at least one of Claims 1-10 wherein 0.1 to 10 percent by weight of the cationic silicone is adsorbed by the textile.

12. The method according to at least one of Claims 1-11 wherein the treatment provides antimicrobial properties to the textile.